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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF :
HIDEKI ISHIHARA, ET AL. : EXAMINER: DOUYON, LORNA
SERIAL NO: 10/523,814 :
FILED: FEBRUARY 4, 2005 : GROUP ART UNIT: 1796
FOR: FRAGRANCE COMPOSITION :

DECLARATION UNDER 37 C.F.R. §1.132

COMMISSIONER FOR PATENTS
ALEXANDRIA, VIRGINIA 22313

SIR:

Now comes Ms. Takami Gema, who deposes and states:

1. I am a named co-inventor of the above-identified application.
2. I am a graduate from Toho University and received my bachelor's degree in the year 1992 majoring pharmacy.
3. I have been employed by Kao Corporation, the assignee of the above-identified application for 18 years as a researcher in the field of perfumery chemistry.
4. The following experiments were performed by me or under my supervision.
 - A. In a first experiment, the compositions in Table 1 were prepared.

Table 1.

(wt.%)	Test Example 1	Test Example 2	Test Example 3	Test Example 4
Sodium POE(2) lauryl ether sulfate	11	1	11	11
Sodium lauryl sulfate	5	0.5	5	5
Cationized guar gum	0.3	0.3	0.3	0.3
Citric acid	0	0	0	0
Malic acid	0.75	0.75	0.75	0.75
Lactic acid	0.1	0.1	0.1	0.1
Sodium chloride	0.2	0.2	0.2	0.2
Benzyl alcohol	0.5	0.5	0.5	0.5
Cocoyl monoethanolamide	1	1	1	1
Dimethicone (viscosity: 100,000 cps)	0.5	0.5	0.5	0.5
Amodimethicone	0.1	0.1	0.1	0.1
Myristyl alcohol	1	1	1	1
Cetanol	0.5	0.5	0.5	0.5
Ethylene glycol distearate	3	3	3	3
Cationized hydroxyethylcellulose	0.3	0.3	0.3	0.3
Glycerol	1	1	1	1
Sodium hydroxide	q.s. to pH below			
Deionized water	Balance	Balance	Balance	Balance
pH	4	4	5	6
Acidic odor	Strong	Little	Strong	Little

For each of the compositions, the odor was assessed by sniffing head space of glass bottles in which each samples placed with the above results. These 110-ml bottles were 9.5 cm tall with 3.0 cm i.d. opening and 5.0 cm i.d. wide bodies. Theses bottles were checked odorless preliminary to the sniffing test. Fifty ml of the sample was placed in the one bottle before putting the lid on. The bottled were allowed to return to room temperature for about 6 hours, then opened and sniffed by a panel of 3 experts.

As shown by the results presented in Table 1, the composition having pH of 5 (Test Example 3) has strong acidic odor, while the composition having pH of 6 (Test Example 4) does not have strong acidic odor.

As also shown by the results presented in Table 1, the composition which contains 16 wt.% of the anionic surfactant (Test Example 1) has strong acidic odor, while the composition comprising 1.5 wt.% of the anionic surfactant (Test Example 2) does not have strong acidic odor.

B. In a second experiment, the compositions in Table 2 were prepared.

Table 2.

	Test Example 1	Test Example 5
	Weight%	Weight%
Sodium POE(2) alkyl ether sulfate	11.0	15.0
Sodium lauryl ether	5.0	
Cationized guar gum	0.3	
Citric acid		0.5
Malic acid	0.75	
Lactic acid	0.1	
Sodium sulfate		1.0
Sodium chloride	0.2	
Benzyl alcohol	0.5	0.5
Cocoyl monoethanolamide	1.0	3.0
Dimethicone (viscosity: 100,000 cps)	0.5	
Amodimethicone	0.1	
Myristyl alcohol	1.0	
Cetanol	0.5	
Ethylene glycol distearate	3.0	2.0
Cationized hydroxyethyl cellulose	0.3	1.0
Glycerol	1.0	
Sodium hydroxide(48% aqueous solution, pH adjuster)	q.s. to pH 4	q.s. to pH 4
Deionized water	Balance	Balance
Total	100.0	100.0

Odor intensity and property after storage of 50 °C, 2-week	3.5	3.5
	increasing sweet and acidic odor	increasing sweet and acidic odor

For each of these compositions, the odor was assessed by the following method.

Fifty ml of the test sample was placed in the one glass-bottle before putting the lid on. These 110-ml bottles were 9.5 cm tall with 3.0 cm i.d. opening and 5.0 cm i.d. wide bodies. These test samples placed in the closed glass bottles were stored for 2 weeks in constant-temperature chambers controlled at 0 °C and 50°C, respectively. After the temperature of the respective samples were allowed to return to room temperature 2 weeks later, the odor assessment was performed by sniffing head space of glass bottles in which each samples placed. The odor intensity from the sample stored at 50°C was ranked in comparison with

the odor intensity from the sample stored at 0°C was taken as standard. The ranking was performed by a panel of 3 experts according to the standard below, and the average of their scores was indicated with 0.5 increments, while the unanimous odor property was desired by the same three panels.

Ranking Standard of Odor Intensity:

- 5: Substantially the same compares with the standard (the sample stored at 0°C).
- 4: Slightly changed compared with the standard.
- 3: Changed compared with the standard.
- 2: Obviously changed compared with the standard.
- 1: Pronouncedly changed compared with the standard.

As shown by the results presented in Table 2, the compositions of Test Examples 1 and Test Example 5 have the same odor intensity and increasing sweet and acidic odor after storage at 50°C for 2 weeks. In Table 2, the composition of Test Example 1 comprising silicones contains a larger amount of fatty acids such as iso-valeric acid, n-butyric acid and hexanoic acid, which are known to have offensive odor caused by deterioration, while the composition of Test Example 5, which does not comprise silicones, does not contain a large amount of fatty acids. However, the odor characteristics of the compositions of Test Example 1 and Test Examples 5 are similar after storage at 50°C for 2 weeks. Thus, the claimed acidic hair composition masks the offensive odor and has long-term stability.

The results are shown in Table 3 and the following chromatograms. The chromatograms were obtained by Solid Phase Microextraction on the following condition.

Analytical Method:

SPME (Supelco)

Fiber: Divenylbenzene/Carboxene/Polydimethylsiloxane (2 cm 50/30µm)

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Incubation Temp: 40°C
Extraction Time: 100 min
Desorption Time: 10 min
Desorption Temp: 250°C

Instrument:

Gas Chromatograph: Agilent 6890
Mass Spectrometer: Agilent 5973 MSD

GLC Condition

Column: DB-WAX (J&W) length: 60 m, Diam: 0.25 mm, Film: 0.25 µm
Flow (He) 0.8 mL/min
Injection Mode: Splitless (4 min)

Oven Temp:

Initial Temp: 40°C (Hold Time 4 min)
Rate 1: 6°C/min (Final Temp 1: 70°C)
Rate 2: 2°C/min (Final Temp 2: 240°C)

Mass Spectrometer:

Ionization Method: Electron Impact (70 eV)

Samples 1 and 2 are represented by the chromatograms after storing Test Example 1 containing silicones for 2 weeks at 5 and 50°C, respectively. Samples 3 and 4 are represented by the chromatograms after storing Test Example 5, which does not contain silicones, for 2 weeks at 5 and 50°C, respectively. The results of Table 3 and the following chromatograms show that after storing the samples at 5°C, Sample 3 has a similar or lower content of fatty acids compared to Sample 1. After storing the compositions of Test Example 1 (Sample 2)

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and Test Example 5 (Sample 4) at 50°C for 2 weeks, Sample 4 has significantly greater content of fatty acids, which are known to have offensive odor caused by deterioration, compared to Sample 2. However, the odor characteristics of Sample 2 and Sample 4 are similar because of the masking effect of the claimed acidic hair composition, as shown in Table 2.

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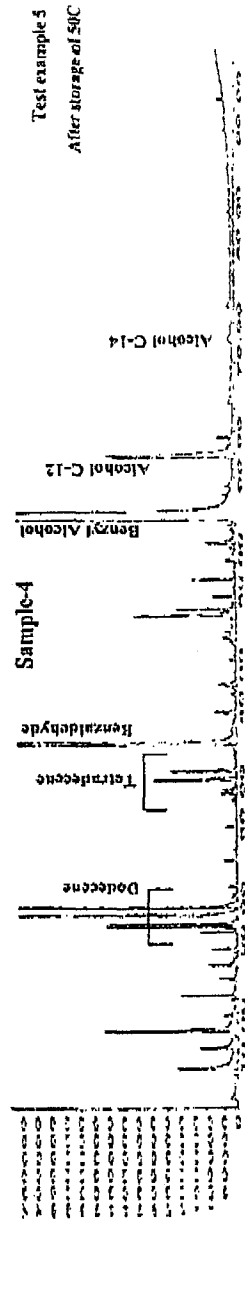
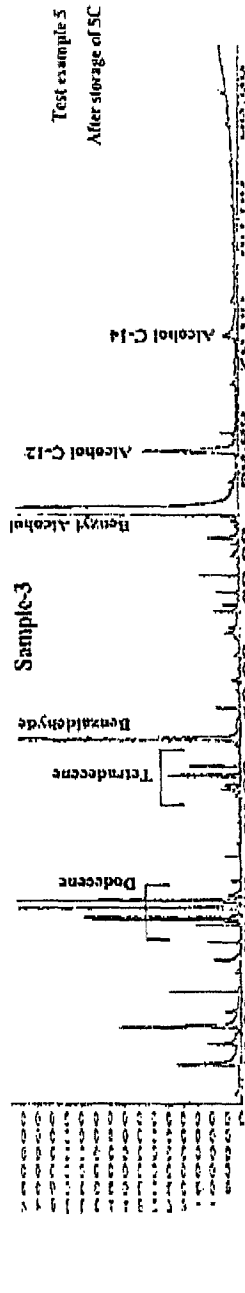
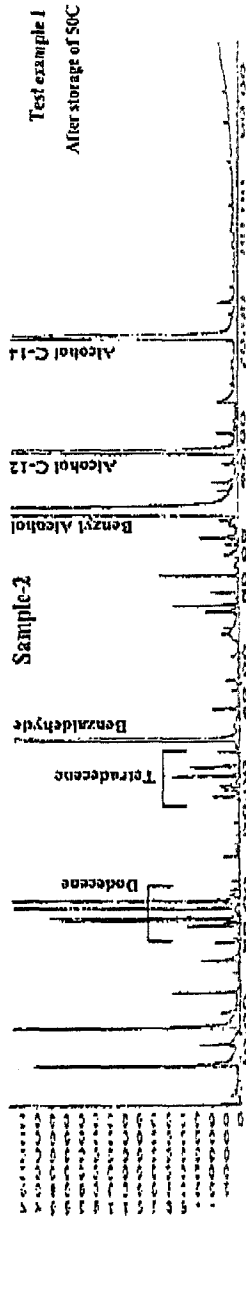
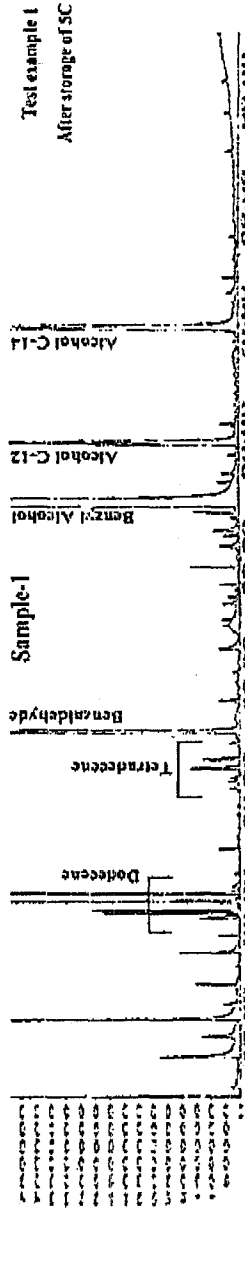
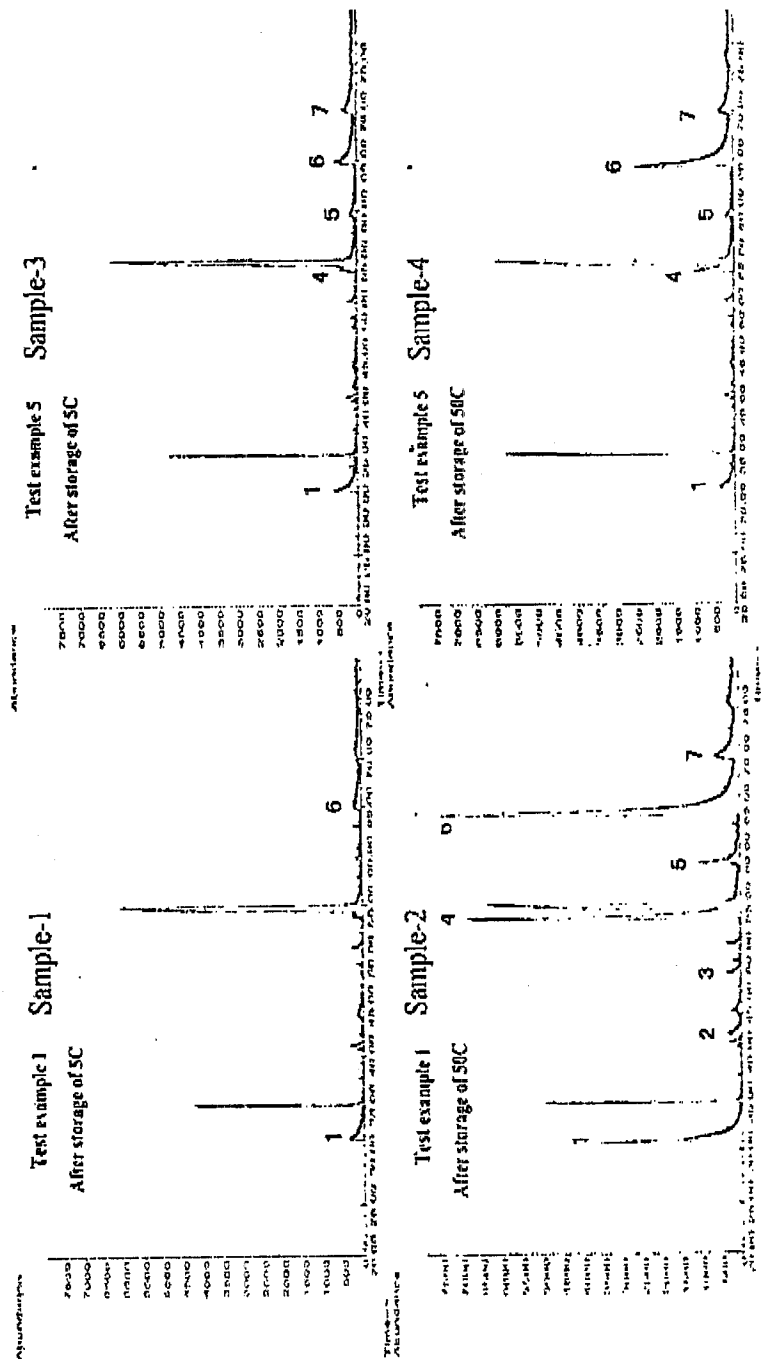


Table 3 Fatty Acid Amount (EX ion: M/Z=60)

No.	Fatty Acid	RT	Sample-1	Sample-2	Sample-3	Sample-4
1	Acetic Acid	31.153	116,310	98,805	217,362	169,607
2	n-Butyric Acid	41.886	-	96,440	-	-
3	n-Palmitic Acid	48.068	-	69,189	-	-
4	Hexanoic Acid	53.689	-	1,260,532	90,130	276,030
5	Heptanoic Acid	59.284	-	138,875	34,935	47,006
6	Octanoic Acid	64.154	93,069	3,375,948	215,249	1,021,229
7	Nonanoic Acid	59.959	-	212,503	138,082	141,230



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5. Thus, the experiments presented above show that offensive odor of a cosmetic base is caused by the low pH, a large amount of surfactants and by the presence of silicones.

6. The undersigned declare further that all statements made herein are of his own knowledge are true and that all statements made on information are believed to be true. Further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

7. Further, Declarant saith not.

Takami Gema
Signature

Oct. 20, 2010
Date